



Date: June 22, 1984

Subject: Primary Metals R&D - Monthly Report - June 1984

From/Location: E. L. Cambridge/TRL

To/Location: J. G. Kaufman/AHR

PRIMARY OPERATIONS

Alumina Characterization

Results from the literature survey work to characterize the impact of alumina properties on smelter operation were presented to Columbia Falls staff. The initial objective of this project is to develop an information matrix illustrating casual relationships. Columbia Falls input was received and will be incorporated into the matrix. The eventual intent is to develop a tool that can be used to determine the technical and economic impact of potential swaps, purchases, etc. The qualitative information matrix will be assembled during August.

MLI Technology

A location for the MLI laboratory equipment has been determined. Materials are now on site for the slow-bake furnace and construction will be complete within a month. Preparation of coke for initial testing has begun. This project remains on schedule for January 1985 completion, and will provide the capability to duplicate Mitsubishi's laboratory evaluation and quality control procedures.

Technical Exchange - Reilly Tar and Chemicals

A meeting with Reilly personnel was held on June 6 in Indianapolis, to exchange information on the effect of pitch properties on carbon anode performance in a Hall cell. Raouf Loutfy and Syver Hellem from Harvey Labs attended with Columbia Falls personnel.

In general, Reilly indicated that, in their view, it is the pitch user's responsibility to define their needs regarding pitch properties. It is our assessment that Columbia Falls qualitative observations coupled with Harvey's reactivity test set-up and our own anode test facility are complimentary programs which should result in a better definition of pitch and coke specifications for Columbia Falls. We intend to continue to work cooperatively in this area.

ADVANCED MATERIALS OPERATIONS

AD-123 Process

Development activities to support final process operating conditions and configuration continued. A correlation between lab milling test conditions and dry milling in the 54" x 42" pilot mill has been established. This will allow product milling characteristics to be determined in 4 hour lab runs instead of 28 hour pilot mill runs. A test matrix has been designed to evaluate ceramic properties and milling characteristics for the complete CERALOX 95 range of surface areas, which will be carried out in the lab equipment.

Equipment is being modified to perform a series of crystallization tests in the 40°-90°C range to define the effect of temperature on crystallite size, shape, and ultimately physical and ceramic properties of finished product. The work will define the optimum operating temperature for the plant crystallizer.

NEW PRODUCT/PROCESS OPPORTUNITIES

Enzyme Immobilization Supports

Samples of Corning controlled pore glass enzyme supports have been obtained. Analysis of these forms will provide insight into the specific requirements needed to fabricate specialty alumina supports.

Sol Gel

A sample of Conoco "Catapal" alumina has been received for use in studies to determine if PCACH is an acceptable/superior substitute.

Magnesium Production Process

(a) HCl Acid Process Option

Specific acid-leach conditions have been determined which significantly improve selectivity with respect to Fe and Al but not Ca. It appears that acid leach alone will not give adequate Mg-Ca separation. Work is in progress to obtain relevant solubility data for chlorine solutions to evaluate the effect of a leach-liquor sulfate addition to precipitate CaSO_4 . This or other process options may provide adequate Mg-Ca separation.

Acid leaching of uncalcined magnesite of reflux has proved feasible but frothing from CO_2 evolution is excessive. Whether there is an economic advantage to this method, which bypasses the ore calcination step will be determined in subsequently planned evaluations.

(b) Carbonation Process

Work began this month on the second proposed flowsheet for Mg recovery from magnesite. Magnesite was calcined, rehydrated, and extracted with carbon dioxide solution. The selectivity of magnesium extraction versus that of impurities was good. A trial sample of recovered $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ exceeded the purity of commercial MgO on an equivalent basis.

However, in these initial trials, extraction efficiency for Mg has been below 50%. Experiments are underway to determine the cause of the fall-off in Mg extraction rate. There are several possible explanations and low recovery in the first experiment is by no means cause for discouragement.

AD-120 PROGRAM

PCACH Technology

(a) ACH Crystallization

The volume shape factor for ACH crystals has been determined to be 0.65. This factor will be used in modelling the crystallization process using population balance theory to determine commercial crystallizer settling design and operating conditions.

Continuous operating campaigns were run to provide material for chlorination tests and to further confirm operating conditions. The Struthers design capacity of 12 lbs/hr for the lab crystallizer was reconfirmed.

(b) Calcination

Experiments to characterize the effect of the type of calcination technology (batch rotary versus continuous fluid bed) on PCACH properties, particularly residual chlorine were carried out. Since Alcoa has now confirmed that HCl from residual chlorine in the PCACH is the chlorination catalyst, this characteristic is of paramount importance. It was found that equivalent residual chlorine was obtained in a continuous fluid bed at approximately 50°-100°C lower temperature than the batch rotary process. This lower potential processing temperature is advantageous as it relaxes materials of construction requirements somewhat. We need to get the same data for a continuous rotary kiln to complete the comparison. As we do not currently have this equipment, consideration is being given to having Ruthner perform these tests.

(c) Corrosion Testing

This program has been planned and resources allocated. Aqueous phase testing will begin June 25. High temperature gas phase testing will start July 1.

REACTOR TECHNOLOGY

(a) CxCly Destruction

Having now established technical feasibility, a conceptual flowsheet, material and energy balance for CxCly destruction with hydrogen, after the cell drop-out box is being developed. This process will be reviewed with Alcoa and compared with their proposed method to develop a recommendation to the joint management committee for which approach to take for commercial demonstration.

(b) High Pressure Fluid Bed Reactor

Based on Dye's predictions of the behavior of off-gas species and direct input from Alcoa, the HPFB reactor has been redesigned to effectively recycle entrained solids and accumulate impurities in the appropriate components of the off-gas train. The two major modifications are (1) bottom feeding of PCACH to eliminate impurity accumulation in the original, cold, top-feeding tube and (2) an internal filter to recycle entrained solids at the correct temperature. These modifications, currently in progress, should be completed during July. They have been incorporated into a restructured project plan, giving a new target date of August 1 for the start of the 100 hour chlorination run. There is no projected negative impact on the overall schedule as a result of these changes.

(c) Dye Contract

At our request, Professor Dye has submitted a proposal to expand his program to include the impact of V, Na, and S impurities from coke. The cost is an additional \$32,000 which is available in the overall budget. Stan Becker has been asked to draft an appropriate contract revision/addition.

FACILITIES

SEM/EDX

A Coates and Welter field emission scanning electron microscope with attached energy dispersive x-ray unit is being transferred from ARCO Solar's Bioengineering group to our facility. This will provide a valuable capability for our CERALOX activities as well as several other research projects.

Project Planning

As Denver is planning to discontinue support of PROJECT/2 software in 1985. We are investigating possible alternatives, one of which is a stripped down version of PROJECT/2 called QWIKNET which can operate on our PC's.

REPORTS AND PUBLICATIONS

The following reports were issued:

- 84-TP-4 "Applications of Inorganic Materials as Supports for Immobilized Enzymes or Cells" by D. M. Blake.
- 84-TP-5 "AD-120 Process-Progress Report No. 5 - The Effect of Coke Type and Pretreatment on Performance in the Chlorination of PCACH" by M. Curran-Hays, S. Young, and R. O. Loutfy.
- 84-TP-6 "AD-120 Process-Progress Report No. 6 - The Effect of Chlorination Conditions on Chlorinated Hydrocarbon Formation in the Production of Aluminum Chloride" by M. Curran-Hays, S. Young, and R. O. Loutfy.
- 84-TP-7 "AD-120 Process-Progress Report No. 7 - The Effect of Calcination Conditions upon the Properties of Rodeo Collier Coke" by K. R. Weisbrod.
- 84-TP-8 "Sebree Anode Formulation Optimization Phases I and II" by K. R. Weisbrod and I. Vogelmann.

PERSONNEL

Computer Training

A local, one day personal computer training course covering IBM PC disk operating system and LOTUS 123 software was attended by 12 of our staff.

Other Training

E. L. Cambridge attended the ARCO Metals Management Development Seminar.


J. C. Withers will be attending B. Karasek's Communications in Management (aka Interpersonnel Awareness) course in Chicago, June 27-29.

Headcount

Mike Burrell joined the Carbon group as an Associate Technician on June 11. Mike had worked for us as a temporary earlier this year and has 4 years of previous experience with Phelps Dodge, Asarco, and UOP.

As a result of this change, current permanent staff breakdown is:

Managers	4
Professionals	12
Technician	17
Clerical	1
Total	<u>34</u>



E. L. CAMBRIDGE

ELC:dg

cc: D. M. Blake/TRL
J. E. Davis/TRL
R. O. Loutfy/TRL
D. S. Moran/TRL
J. C. Withers/TRL
R. W. Bartlett/TRL
S. Maitra/AH
R. J. F. Thorpe/AHR
H. Rack/AHR
T. E. Fine/AH
R. L. Williams/AHR
J. A. Roberts/AHR